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# Self-templated synthesis of polycrystalline hollow aluminium nitride nanospheres

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## Index:

SI 1. Characterization of the ball-like Al particle precursor.

SI 2. Evolution of the nitridation product with temperature.

SI 3. Raman spectrum of the hollow AlN spheres.

SI 4. Photoluminescence spectrum of the hollow AIN spheres.

For testing the reasonability of the proposed growth mechanism, Al particles with ball-like morphology were also used as a precursor to synthesize the hollow AlN nanospheres in a similar process. The precursor and products were characterized by TEM, XRD, Raman and PL spectra. The results support the proposed growth mechanism as shown below:

#### SI 1. Characterization of the ball-like Al particles precursor.

The ball-like Al particles were fabricated by arc discharge, purchased from Siping Gaosida Nano Material Co., Ltd., China. The diameters of the particles are about 100–200 nm (Figure S1a) and the purity of the precursor is quite good (Figure S1b).



Figure S1: TEM image (a) and corresponding XRD curve (b) of the ball-like Al particles.

#### SI 2. Evolution of the nitridation product with temperature

The experimental process is the same as described in the main text but only NH<sub>3</sub> was used since the precursor was pure enough. Figure S2 shows typical TEM images of the as-products for different reaction temperatures. It is seen that a large portion of the ball-like Al particles was changed to hollow spheres of about 200 nm in diameter and 10 to 20 nm in shell thickness, especially when the reaction temperature was higher than 1000 °C. These hollow spheres are h-AIN as reflected by the XRD curves (Figure S3). It is seen that, with increasing temperature, the TEM images and the XRD curves of the products show similar changes to those in the main text. It is also learnt that the formation of the hollow h-AIN nanospheres started at around 800 °C and was complete at around 1200 °C.



Figure S2: Typical TEM images of the products produced by nitriding ball-like Al particles at different temperatures. (a) 800 °C; (b) 1000 °C; (c) 1200 °C; (d) 1300 °C.



Figure S3: Corresponding XRD curves of the products in Figure S2.

### SI 3. Raman spectrum of the hollow AIN spheres.

The Raman spectrum of the hollow AlN spheres obtained at 1300 °C (Figure S4) shows six Raman-active modes, i.e.  $1A_1(TO) + 1A_1(LO) + 1E_1(TO) + 1E_1(LO) + 2 E_2$  (high). The possible reason for the weakening of  $A_1(TO)$  and  $E_1(TO)$  modes were discussed in the text.



Figure S4: Raman spectrum of the hollow AlN spheres.

### SI 4. Photoluminescence spectrum of the hollow AlN spheres.

The PL spectrum of the hollow AlN spheres (Figure S5) shows three emission peaks, i.e. 409 nm (3.04 eV), 456 nm (2.72 eV), 540 nm (2.30 eV). The possible emission mechanism was speculated in the main text.



Figure S5: Photoluminescence spectrum of the hollow AlN spheres.